# DRAFT: The Potential for Generating Inadvertent PCBs through $TiO_2$ Manufacturing Using the Chloride Process

DRAFT

A white paper prepared for the Spokane River Regional Toxics Task Force

*DRAFT – FOR REVIEW*

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# 1. Executive Summary

The Spokane River Regional Toxics Task Force (SRRTTF) leads efforts to find and reduce toxic compounds in the Spokane River with an emphasis on polychlorinated biphenyl (PCB) compounds. The SRRTTF contracted Northwest Green Chemistry (NGC) to produce a white paper/memo to learn more about the production of titanium dioxide ($TiO_2$) and its potential to produce inadvertent PCBs that could end up in consumer and industrial products that could impact the Spokane River. NGC gathered information via reviews of the scientific literature, performed web-based research of trade publications, government publications, and company websites, and interviewed some academic and industry experts.

There is limited evidence that $TiO_2$ manufactured using the chloride process may produce low levels of PCBs that could be associated with the $TiO_2$ product. It is not possible to say if these PCBs are at a level of concern until more is known about the range of levels produced, the congeners formed, how the product is used, and how people are exposed. Expert opinion in industry generally holds that PCBs are not expected to be present because the chloride process includes very high temperature unit processes that should destroy organic compounds like PCBs. Therefore testing is not done, and test data are not readily available. However, one pigment and coatings supplier who purchases $TiO_2$ for use in products shared results from testing two batches of $TiO_2$ powder from two different manufacturers, and found PCB levels at 85ppb using Method 1668C. Experts also noted that chloride manufacturing processes can vary between companies based on the original technology used, and process improvements made to it over time. This variability may result in different degrees of PCB destruction.

We believe it is not accurate to say that inadvertent PCBs 1) cannot be formed and/or 2) are formed but completely destroyed during high temperature manufacture using the chloride process. Information from the literature and limited test data suggest sufficient evidence to warrant further investigation. We recommend testing $TiO_2$ manufactured by different suppliers who use the chloride process. We recommend testing the pure pigment first, rather than formulated products containing $TiO_2$ because $TiO_2$ could pick up PCBs from the ambient environment; and other chemicals used in a formulation could contain PCBs. We also recommend testing both pigmentary and ultrafine (nano) $TiO_2$. Ultrafine $TiO_2$ typically undergoes different process steps to control for particle size. Given the scale of production of $TiO_2$ globally using the chloride process and the dominance of the chloride process in the US, $TiO_2$ production may be a non-trivial contributor to emissions of inadvertent PCBs.

## 2. Introduction

The Spokane River Regional Toxics Task Force (SRRTTF) is a multi-stakeholder initiative organized to lead efforts to find and reduce toxic compounds in the Spokane River (SRRTTF 2018). The goal of the task force is to develop a comprehensive plan to bring the Spokane River into compliance with water quality standards for PCBs (polychlorinated biphenyls). These pollutants exceed water quality standards in several segments of the river.

The SRRTTF is actively working to better characterize the amounts, sources and locations of PCBs and other toxics entering the Spokane River; preparing recommendations to control and reduce those sources, reviewing Toxic and Source Management Plans, developing performance-based limits, and monitoring and assessing the effectiveness of toxics reduction measures.

The SRRTTF commissioned Northwest Green Chemistry (NGC) to produce a white paper/memo to learn more about the production of titanium dioxide ($TiO_2$) and its potential to produce inadvertent PCBs (iPCBs) that could end up in consumer and commercial products that could eventually impact the Spokane River. NGC gathered information via reviews of the scientific literature and performed web-based research of trade publications, government publications, and company websites. This work was supplemented with interviews of some academic and industry experts. See Appendix 1.

## 3. Production of Titanium Dioxide ($TiO_2$)

Approximately 6 million metric tonnes (6.6 million US tons) of the pigmentary $TiO_2$ are produced annually worldwide and this number continues to increase. $TiO_2$ pigment is a global commodity with consumption distributed between paints (including lacquers and varnishes) (60%), plastics (25%), paper (3%), and other (4%). (Maas 2018). Other uses of $TiO_2$ included catalysts, ceramics, coated fabrics and textiles, floor coverings, printing ink, and roofing granules. Other industry estimates are lower for paints but the relative ratio to plastics and paper is the same. The top five global $TiO_2$ vendors in 2017 were Chemours, CRISTAL, Huntsman, KRONOS and Tronox (Business Wire 2017). China is the primary manufacturer (2.94 million tons) followed by Europe, followed closely by the US (1.36 million tons) (US Geological Survey 2018). Ultrafine (nano) TiO2 is estimated at about 1% of $TiO_2$ production and manufacturers of pigmentary $TiO_2$ do not necessarily also manufacture the ultrafine (nano) form.

There are two primary ways to process titanium containing minerals (ilmenite or rutile) into intermediates that are then oxidized into $TiO_2$. The 'sulphate' method involves digesting the mineral in sulphuric acid to generate titanyl sulphate. The titanyl sulphate is then hydrated to form the solid titanium dioxide. The hydrated titanium dioxide is then heated in a furnace ((300K-1000K) depending on the process step, and the desired end crystal structure) to produce anhydrous titanium dioxide. Finally the titanium dioxide is milled down to the desired particle size before being used as a pigment (Kienast 1973) (Essential Chemical Industry 2017). See (Figure 1).

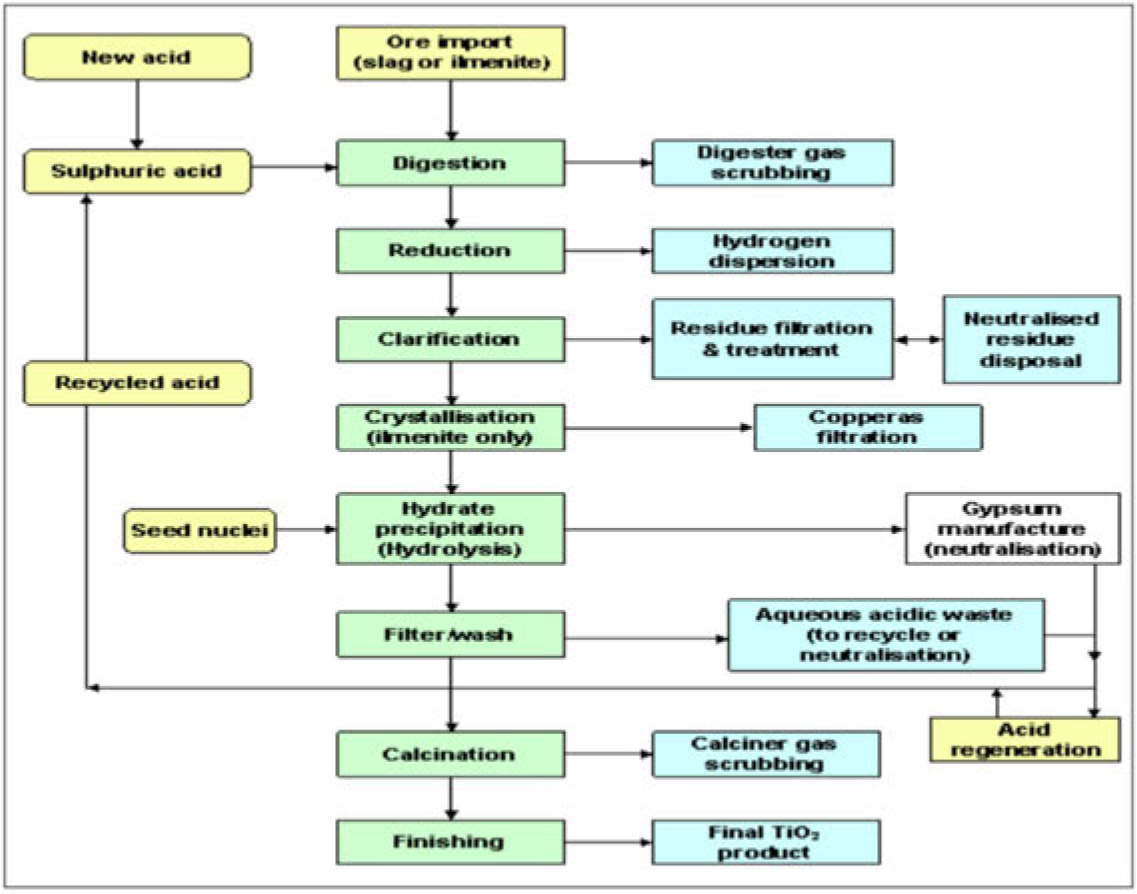


**Figure 1. Outline flow diagram – titanium dioxide production by the sulphate process route (European Commission 2007)**

The 'chloride' method involves reacting the raw mineral with chlorine gas at high temperatures (>1200C) in the presence of coke to produce titanium tetrachloride. The resultant titanium tetrachloride is then burnt in the presence of oxygen to produce titanium dioxide. During this step nucleating agents are added and reaction conditions are controlled to give the desired particle sizes for the pigments (Figure 2). See also Appendix 2 for an overview of the $TiO_2$ chloride manufacturing process provided by CRISTAL.

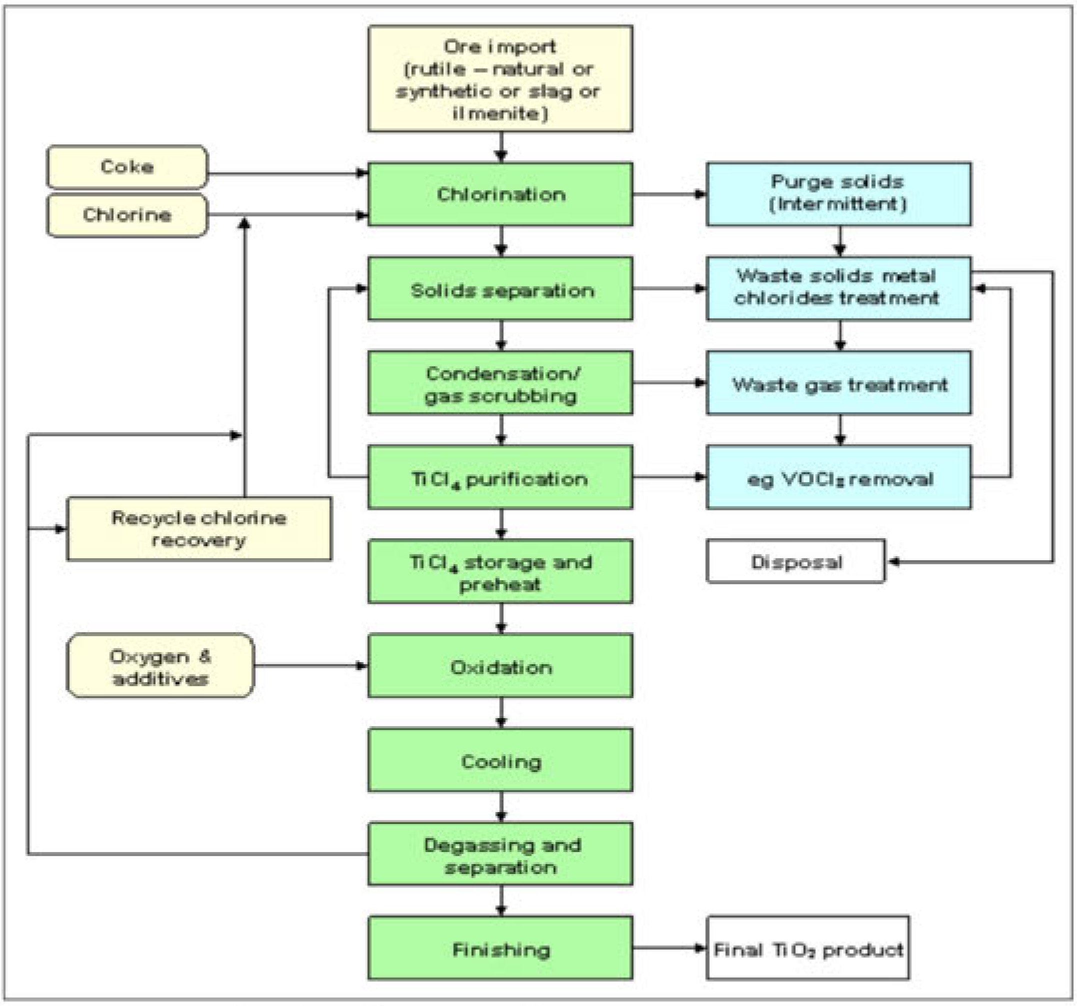


**Figure 2. Outline flow diagram – titanium dioxide production by the chloride process route (European Commission 2007)**

Alternatively, titanium dioxide particles can be produced by hydrolysing the titanium tetrachloride in a controlled manner to create ultrafine $TiO_2$, aka nanoparticles (Cheng 1994). See Figure 3. The difference is due to the use of hydrolysis rather than oxidation for better particle size control.

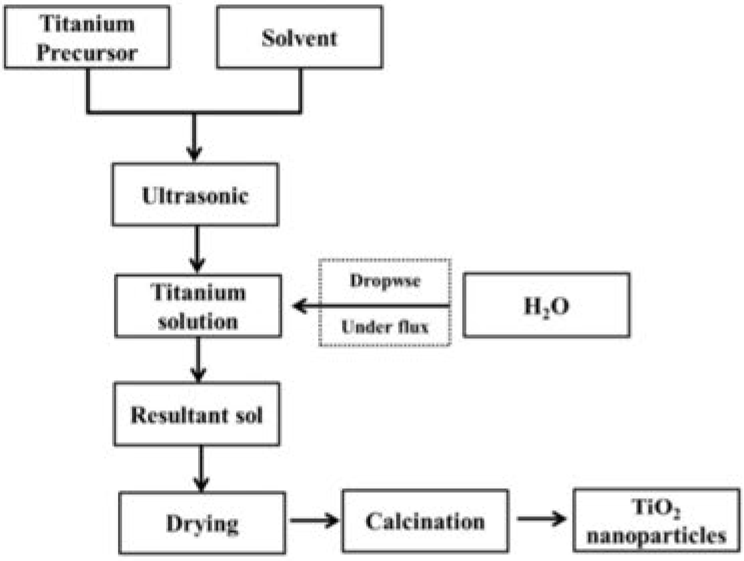


**Figure 3. Scheme of the preparation of TiO2 nanoparticles sol-gel** (Gupta 2012)

## 3.1. Relative Use and Benefits of the Chloride versus the Sulphate Process

The sulfate process is the older process of the two, developed in 1916. The chloride process was developed in 1948. Most industry sources state that the use of the chloride process continues to grow worldwide with the exception of China, where new sulphate plants are being built. Based on web research and expert interviews, it was determined that the primary, if not exclusive, mode of production of $TiO_2$ in the US is the chloride process. The primary mode of production in China is the sulphate process. And in Europe, the relative ratio of use of both processes is mixed.

| Year | Sulphate process (2) | | Chloride process | | Total |
|---|---|---|---|---|---|
| | kt per year | % | kt per year | % | kt per year |
| 1965 | 1254 | 90.3 | 135 | 9.7 | 1389 |
| 1970 | 1499 | 77.4 | 437 | 22.6 | 1936 |
| 1977 | 1873 | 72.3 | 716 | 27.7 | 2589 |
| 1988 | 1781 | 60.2 | 1178 | 39.8 | 2959 |
| 1995 | 1481 | 46.0 | 1739 | 54.0 | 3220 |
| 2000 (1) | 1540 | 40.0 | 2310 | 60.0 | 3850 |

(1) Estimated; (2) A number of plants based on the sulphate process have recently been commissioned in China.

**Table 1. World production of $TiO_2$ pigment using the sulphate vs the chloride process (1965-2000)** (European Commission 2007)

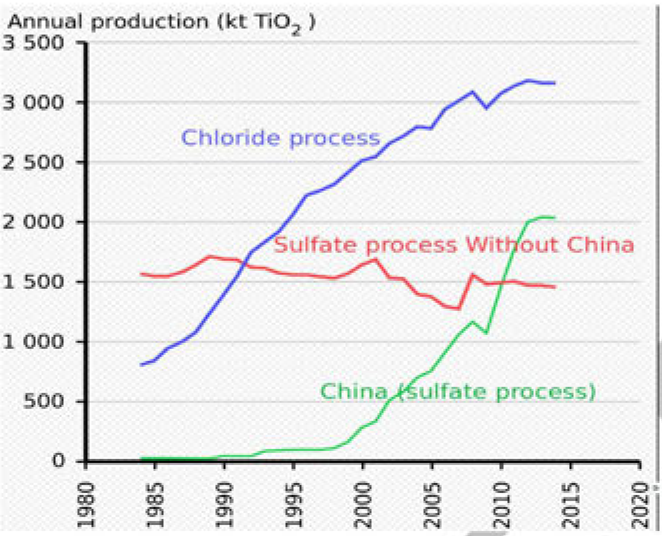


**Figure 4. Annual production of TiO2 pigment using the sulphate vs the chloride process with a focus on China** (Borvan53 2015)

The sulphate process is considered less environmentally friendly because of the acid wastes that are generated. However, this may not fully address issues of toxicity that arise with wastes associated with the chloride process (Essential Chemical Industry 2017).

| Sulfate Process | Chloride Process |
|---|---|
| long established and simple technology | new technology |
| uses lower grade, cheaper ores | needs high grade ore |
| batch process | continuous process |
| large amounts of waste materials | small amounts of waste formed with toxicity problems: $Cl_2$ and $TiCl_4$ |
| pollution control expensive | recovery and recycling of chlorine possible |
| produces anatase and rutile pigments | only produces rutile pigments |

**Table 2. Comparing the sulfate and chloride processes** (Essential Chemical Industry 2017)

## 4. Feasibility of Generating Inadvertent PCBs

Ctistis et al. demonstrated the synthesis and origin of unintentional impurities of chlorinated persistent organic pollutants (POPs) in the manufacture of $TiO_2$ when rutile and/or anatase titanium is reduced with carbon and oxidized with chlorine to produce titanium tetrachloride. (Ctistis 2016) The $TiCl_4$ is distilled and reoxidized mainly in hydrolysis or in a pure oxygen flame or plasma at very high temperatures. The authors propose that it is during this carbo-thermal chlorination process that PCBs and potentially polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are formed. These inadvertent impurities can stay within the generated material and end up being incorporated into the final commercial products. While the focus of the article is on nano $TiO_2$ particles contaminated with POPs, the proposed pathway applies to pigmentary $TiO_2$ production also.

4840 Environ Sci Pollut Res (2016) 23:4837–4843

$$2\,FeTiO_3 + 7\,Cl_2 + 6\,C \xrightarrow{900\,^\circ C} 2\,TiCl_4 + 2\,FeCl_3 + 6\,CO \quad (1)$$

$$TiCl_4 + 2\,H_2O \xrightarrow{\text{hydrolysis}} TiO_2 + 4\,HCl \quad (2)$$

$$TiCl_4 + O_2 \xrightarrow{\text{plasma}} TiO_2 + 2\,Cl_2 \quad (3)$$

$$12\,C + 6\,Cl_2 \xrightarrow{\Delta T} 2\,C_6Cl_6 ; C_{12}Cl_{10} + Cl_2 ; C_{10}Cl_8 + 2\,Cl_2 \quad (4)$$

$$C_6Cl_6 \xrightarrow{\Delta T / h\nu} C_6Cl_{6-x}OH_x ; C_6Cl_{6-x}H_x ; C_6Cl_{6-x}H/OH_x \quad (5)$$

$$C_{10}Cl_8 \xrightarrow{\text{plasma}} C_{10}Cl_{8-z}OH_z ; C_{10}Cl_{8-z}H_z ; C_{10}Cl_{8-z}H/OH_z$$

$$C_{12}Cl_{10} \xrightarrow[\text{catalysis } TiO_2]{\text{sintering/}} C_{12}Cl_{10-y}OH_y ; C_{12}Cl_{10-y}H_y ; C_{12}Cl_{10-y}H/OH_y$$

$$\underset{\text{PCBs}}{C_{12}Cl_{x-y}H_y} + O_2 \xrightarrow[\text{catalysis } TiO_2]{\text{sintering/}} \underset{\text{PCDDs}}{C_{12}O_2Cl_{w-z}H_z} + \underset{\text{PCDFs}}{C_{12}OCl_{w-z}H_z} \quad (6)$$

$x, y = 0 - 10 ; w, z = 0 - 8$

**Fig. 1** $TiO_2$ synthesis and origin of unintentional impurities of chlorinated POPs. $TiCl_4$ is produced by the chloride process, which involves the reduction of titanium oxide ores, typically ilmenite ($FeTiO_3$) with carbon under flowing chlorine at 900 °C (*1*). Around 90 % of the $TiCl_4$ production is used to make the pigment titanium dioxide ($TiO_2$) by hydrolysis (*2*) or direct oxidation in a plasma (*3*). During the $TiCl_4$ formation (*1*), unintentionally high and per-chlorinated benzene, biphenyls, and naphthalenes can be formed (*4*). These POPs can be oxidized with oxygen at higher temperatures, with light in the presence of catalysts, for example $TiO_2$ forming ROS, and oxygen plasma combustion (*5*) into lower chlorinated POPs, hydroxylated ones, dioxins, and furans. Furthermore, PCBs can be oxidized with oxygen at higher temperatures in a sintering process, in the presence of catalysts like $TiO_2$ forming PCDDs and PCDFs (*6*)

**Figure 5. Proposed pathway for synthesizing inadvertent PCBs in producing $TiO_2$** (Ctistis 2016)

## 4.1. Expert opinion

NGC staff met with several industry and academic experts to better understand the potential for forming inadvertent PCBs in the $TiO_2$ manufacturing process. These interviews included experts from three of the five largest global $TiO_2$ manufacturing corporations. See Appendix 1 for the list of interviewees.

All three of the major manufacturers shared that they do not test for PCBs in their $TiO_2$ product because they do not expect to find them, mostly due to the very high temperatures used in production that would cause the PCBs to decompose.

One expert from a major $TiO_2$ manufacturing corporation claimed that PCBs are formed in the chloride process but they are destroyed at the high temperatures used to produce the $TiCl_4$ and potentially also during the subsequent oxidation process. This expert also noted that it is possible for $TiO_2$ pigments once produced and sold, to pick up PCBs from the ambient environment at very low levels since it is very difficult to find any environment that is truly free of PCBs. This expert also noted that PCBs in products that contain $TiO_2$ may also come from other chemicals used in product formulations. Another expert noted that PCBs are being found in silicones used in paints and coatings, the dominant use of pigmentary $TiO_2$. Based on preliminary research, the silicones most likely to contain iPCBs are those synthesized from phenyl siloxanes.

Experts from a second large $TiO_2$ manufacturer claimed that because the $TiO_2$ process is entirely inorganic, it is not possible to produce organic compounds such as PCBs. They recommended that the research team review the EU Commission BREF document (European Commission 2007). The research team reviewed the document released by BREF and found reference to estimated emissions of 0.00041 - 0.00025kg of chlorinated organics per /tonne of $TiO_2$ generated using the chloride process in 1999-2002.

| | kg/tonne 1999 | kg/tonne 2000 | kg/tonne 2001 | kg/tonne 2002 |
|---|---|---|---|---|
| **Discharges to water** | | | | |
| Hydrochloric acid | 16 | 13 | 13 | 12 |
| Titanium (Ti) | 0.8 | 0.6 | 0.5 | 0.6 |
| Suspended solids | 0.6 | 0.5 | 0.4 | 0.5 |
| Manganese (Mn) | 0.6 | 0.6 | 0.4 | 0.5 |
| Iron (Fe) | 0.28 | 0.55 | 0.67 | 0.61 |
| Vanadium (V) | 0.03 | 0.03 | 0.03 | 0.02 |
| Chromium (Cr) | 0.003 | 0.004 | 0.004 | 0.002 |
| Zinc (Zn) | 0.006 | 0.007 | 0.009 | 0.005 |
| Nickel (Ni) | 0.019 | 0.015 | 0.004 | 0.001 |
| Lead (Pb) | 0.011 | 0.009 | 0.002 | 0.000 |
| Copper (Cu) | 0.003 | 0.003 | 0.006 | 0.001 |
| Arsenic (As) | 0.0001 | 0.0001 | 0.0001 | 0.0000 |
| Cadmium (Cd) | 0.00018 | 0.00013 | 0.00012 | 0.00001 |
| Mercury (Hg) | 0.00007 | 0.00001 | 0.00000 | 0.00000 |
| Chlorinated organic compounds | 0.00041 | 0.00027 | 0.00025 | 0.00025 |
| **Emissions to air** | | | | |
| Carbon monoxide | 181 | 116 | 65 | 83 |
| Carbonyl sulphide | 3.2 | 1.9 | 1.3 | 0.4 |
| Nitrogen oxides (as $NO_2$) | 1.2 | 1.3 | 1.2 | 1.2 |
| Particulates | 0.4 | 0.1 | 0.1 | 0.1 |
| Sulphur dioxide | 0.3 | 0.2 | 0.1 | 0.1 |
| Hydrogen chloride | 0.2 | 0.0 | 0.0 | 0.0 |
| Chlorine | 0.00041 | 0.00054 | 0.00050 | 0.00012 |
| Hydrogen sulphide | 0.1 | 0.1 | 0.0 | 0.0 |
| Carbon dioxide (ex process) | 437 | 487 | 729 | 576 |
| Carbon dioxide (ex combustion) | 1304 | 1090 | 1110 | 989 |
| Carbon dioxide (from bought in energy) | 609 | 588 | 561 | 540 |
| **Wastes to land** | | | | |
| Non-hazardous waste | 729 | 785 | 881 | 962 |
| Hazardous waste to land/incineration | 0.5 | 1.8 | 0.9 | 1.8 |
| **Resource consumption** | | | | |
| Water usage $m^3$ | 38 | 32 | 31 | 32 |
| Energy usage GJ | 29 | 26 | 26 | 23 |

**Table 3. Summary of emissions (1999-2002) in the Grantham Works $TiO_2$ plant (chloride process)** (European Commission 2007)

While the term chlorinated organics does not necessarily imply production of PCBs, it does demonstrate that production of chlorinated organic wastes or emissions can occur at least at some point in the chloride $TiO_2$ manufacturing process.

A third manufacturer confirmed that PCBs are generated in the chloride $TiO_2$ manufacturing process and that they may or may not be fully destroyed depending on the technology used by the manufacturer. They shared that while all chloride manufacturing is similar at the conceptual level, each company continues to improve its specific process technologies; and some processes are likely to be better than others for destroying PCBs. Most big changes in process technologies occur around the chlorination and oxidation steps; the steps that are most relevant to PCB production (Ctistis 2016).

Very low levels of PCBs in $TiO_2$ powder were measured by a manufacturer of color pigment and coatings who purchases pigmentary $TiO_2$ for use in other products. This manufacturer had two batches of raw $TiO_2$ powder, produced by two different sources, tested for PCBs using EPA Method 1668C. Both batches were found to contain 0.085 ppm (85ppb) PCBs. Method 1668C is the standard method used in the industry for detecting PCBs (HRGC/HRMS 2010). Prior versions of Method 1668C, which revised the upper recovery limit acceptance criteria for some congeners, were 1668B (January 2009), which changed the single lab acceptance criteria to inter-laboratory criteria and the original 1668A (December 1999) (HRGC/HRMS 2010).

### 4.2. Estimated production of inadvertent PCBs

An estimate of global $TiO_2$ production in 2017 was 6 million metric tonnes. If one conservatively estimates that half of the production is based on the chloride process, then about 3 million metric tonnes of chloride-based $TiO_2$ were produced.

If the $TiO_2$ produced via the chloride process contained 85 ppb PCBs, then the annual production of PCBs would be 3 million metric tonnes x $1/1x10^9$ which is 0.255 tonnes (0.288 US tons, 576 pounds) of PCBs.

## 5. Summary and Recommendations

There is some evidence that $TiO_2$ manufactured using the chloride process may produce low levels of PCBs that may be associated with the product. It is not possible to say whether or not these PCBs are at a level of concern until more is known about what the levels are, what the congeners are, how the product is used, and how people and the environment are exposed. While expert opinion in industry holds that it is not necessary to test for PCBs because they are not expected to be present, there is need for testing to better understand the concentration ranges of PCBs that may be formed. Based on our findings to date, we expect that very low levels will be found. With only two test data points (both reporting 85ppb) available at this time, we are not able to define the relevant ranges that can be expected. Expert opinion posits that some chloride technologies will result in negligible (non detectible) levels of PCBs. However, we believe it is not accurate to say that inadvertent PCBs 1) cannot be formed and/or 2) are formed but completely destroyed during high temperature manufacture using the chloride process.

This report is intended to lead to better understanding of the current levels of PCBs that are formed via chloride production of $TiO_2$, and the ability of industry to keep PCB levels to negligible levels through advanced technology. We believe there is limited but sufficient evidence from the scientific literature, expert interviews and the results of analytical testing by a manufacturer of color pigments and coatings to warrant further investigation. We recommend testing both pigmentary and ultrafine (nano) $TiO_2$ for inadvertent PCBs to confirm if, and how much, inadvertent PCBs are produced using the chloride process. Because ultrafine $TiO_2$ is typically produced with hydrolysis instead of oxidation for better particle size control, testing should be done separately for both forms. Given the scale of production of $TiO_2$ globally using the chloride process and the dominance of this process in the US, $TiO_2$ production may be a non-trivial contributor to emissions of inadvertent PCBs. We recommend testing the pure pigment first, rather than formulated products containing $TiO_2$ because $TiO_2$ could 1) pick up PCBs from the ambient environment, and 2) other chemicals used in the formulation could contain PCBs. By testing the pure $TiO_2$ produced by a range of manufacturers, using their proprietary process technologies, it may be possible to resolve the question of the degree to which pigmentary and ultrafine (nano) forms of $TiO_2$ may contain inadvertently generated PCBs.

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# 7. Appendix 1: Individuals Interviewed

**University:**

Rutgers University: Lisa Rodenburg

**$TiO_2$ Manufacturers:**

CRISTAL; Doug Hermann; Mark ??

KRONOS: Kevin Lombardozzi, Olaf Schulze

Chemours: Michael Ober

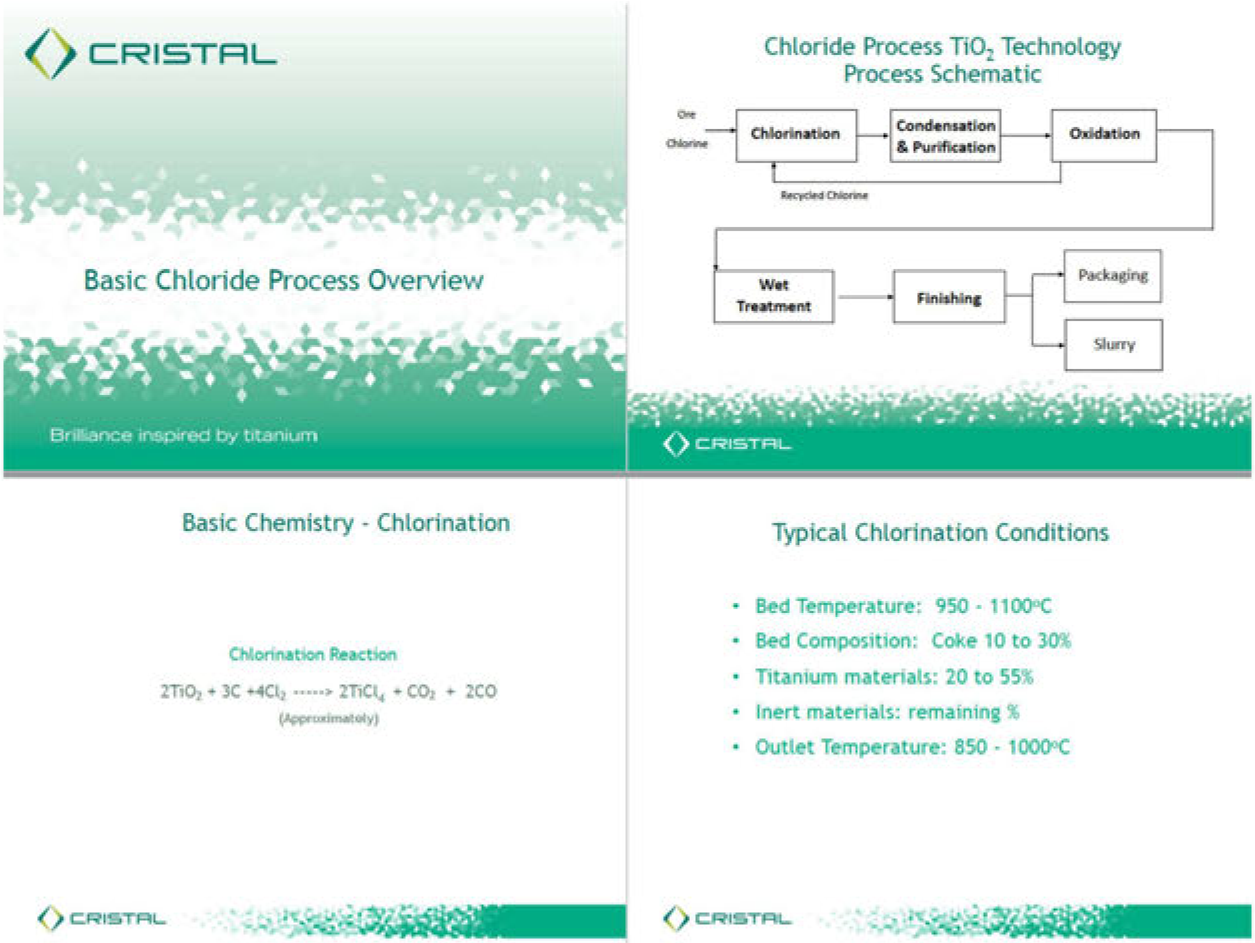
**Pigment and Coating Manufacturers:**

Clariant: Romesh Kumar

Color pigment and coating manufacturer (anonymous)

# 8. Appendix 2: Basic Chloride Process Overview

From non proprietary slides provided by CRISTAL



## Condensation & Purification - Purpose

- Cools the gas stream
- Condenses gaseous TiCl4
- Recovers and collects liquid TiCl4
- Removes solids from Main Process
- Segregates crude TiCl4 streams
- Removes entrained solids
- Removes metal chlorides (Fe, V, Zr, Sn)
- Removes Vanadium ($VCl_4$ & $VOCl_3$)

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## Oxidation - Purpose

- React $TiCl_4$ with Oxygen
  $TiCl_4 + O_2$ -----> $TiO_2 + 2Cl_2$
- Produce pigmentary $TiO_2$
- Release $Cl_2$ for recycle to chlorination
- Separate $TiO_2$ product from recycle gas stream
- Produce $TiO_2$ slurry for Finishing

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## Typical Oxidization Conditions

- $TiCl_4$ vaporizer discharge temp: 400°C
- $O_2$ preheater discharge temp: 950°C
- $AlCl_3$ discharge temp: 450°C
- "Wet air" temp: 1450°C

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